BRISTOL ChemLabS

A CENTRE FOR EXCELLENCE IN TEACHING AND LEARNING

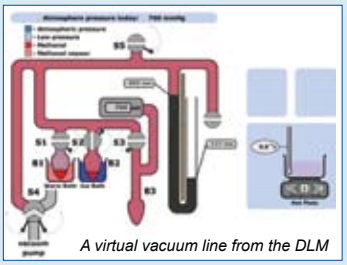
University of BRISTOL

SCHOOL OF CHEMISTRY

# FIRST YEAR UNDERGRADUATE LABORATORY COURSE

First year experiments run for 3 hours, which the students undertake once a week for 24 weeks. Students will be expected to complete pre-lab work before they arrive at the laboratory itself, which will consist of several components:

1. An introduction to the experiment so that the students understand what it is that they will be doing;
2. An online assessment to examine how well they understand the aims of the experiment;
3. An online safety test.



*A virtual vacuum line from the DLM*

Assessment for each experiment will be based upon the pre-lab work, in-lab assessment by demonstators and staff, and in some cases assessment of a post-lab writeup. It is the intention that full writeups are not a feature of the laboratory experience in the first year for every experiment.

## List of Experiments

- Oxidation States of Tin Compounds: Tin (IV) Iodide and Tin (II) Chloride
- Stoichiometry: Preparation and Spectroscopic Analysis of a Copper-Glycinate Complex
- Recrystallisation in the Purification of *trans*-Stilbene
- The Reaction of Benzylamine with Benzoyl Chloride
- Acid/Base Equilibria: The Use of Indicators and pH Meters to produce Titration Curves
- Thermochemistry in the Decomposition of Zinc Carbonate
- The Preparation of Transition Metal Acetylacetonate Complexes
- Stability Constants of Silver Complexes with Amines
- The Reduction of Benzophenone to Diphenylmethanol
- Separation and Extraction: The Use of a Separating Funnel and Using a Basic Solution to Extract Benzoic Acid
- Chemical Kinetics I – Diazonium Salt Decomposition
- The Viscosity of Gases
- The Preparation of some Four-Coordinate Nickel Complexes
- The Resolution of a Racemic Mixture of Transition Metal Complexes
- Esterification and Distillation: The Preparation of Isoamyl Acetate
- Aldol condensation: Dibenzalacetone
- The Enthalpy of Combustion of an Alcohol
- The 2 $NO_2 \rightleftharpoons N_2O_4$ Equilibrium
- Magnetochemistry of Several Metal Complexes Prepared in Earlier Experiments
- The Chemistry of some Compounds of Boron
- The Dehydration of *tert*-Amyl Alcohol
- The Vibrational-Rotational Spectrum of Germane and UV Spectroscopy: 'Particle in a Box'
- Chemical Kinetics II – The Hydrolysis of Ethylformate
- The Enthalpy of Vaporisation of Heptane and Propanol from Flow Calorimeter Measurements
- The Enthalpy of Vaporisation of Methanol from Vapour Pressure Measurements

Following the success of the bid to HEFCE to become a CETL (Bristol ChemLabS), the School of Chemistry at the University of Bristol has undertaken a complete review of its undergraduate laboratory content. Thus we have reconsidered not only the skills we want students to learn, but also our assessment methods and the way postgraduates demonstrate. Moreover, the practical courses have been considered as an integrated whole rather than as disparate inorganic, organic and physical parts.

Much of the learning for a given experiment is to be shifted to before the laboratory practical itself so that students better understand the chemistry they are about to undertake. This will be achieved via the Dynamic Laboratory Manual (DLM), an interactive Web-based system, containing all the information required to carry out the experiment. Students will take an online assessment about the experiment before the start of the lab as well as an online safety test. Some of these tests will be 'scenario-based' and require students to fully understand any hazards in order to decide the best course of action in the event of an accident.

More importantly, we plan to do much more in-lab assessment rather than assessed post-lab write ups. This face-to-face contact will provide a more accurate assessment of the student and provide a much greater opportunity for individual education and feedback.

Dr PJ Wyatt,
Bristol ChemLabS Director and
School of Chemistry Director of
Undergraduate Studies

*Bristol ChemLabS • School of Chemistry • University of Bristol • Cantock's Close • Bristol • BS8 1TS*
*tel +44 (0117) 331 7680 • fax +44 (0117) 925 1295 • chem-cetl@bristol.ac.uk • www.chemlabs.bristol.ac.uk*

# PROPOSED NEW SECOND YEAR UNDERGRADUATE LABORATORY COURSE

Second year students will carry out experiments for 6 hours per week for 24 weeks. This will be preceded at the end of the first year by an intensive session of 7 hours per day for 8 days that will run over a two week period.

Assessment practices and a Dynamic Laboratory Manual will be developed in parallel with the First Year Laboratories.

## List of Experiments

### Intensive Summer Laboratories

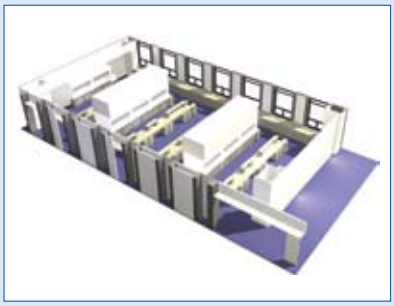
- The use of Grignard Reagents: The Preparation and Reactions of Triphenylmethanol
- The Reduction of 2-Benzofuranylmethyl Ketone using $NaBH_4$ and a Carrot
- An Introduction to Chromatography: Extraction and Analysis of Caffeine
- The Synthesis and Characterisation of a 'High'-Temperature (77K) Superconductor
- The Synthesis of Chromium(II) Acetate: An Introduction to Inert Atmosphere Synthesis
- Calculation of Molecular Structure, Energetics and Spectra using Gaussian
- Scientific Writing: Keeping a Good Lab Notebook
- An Introduction to Electrochemistry

### Second Year Course

- A Thiazolium Catalyst and Stereochemistry in the Synthesis and Reduction of Benzoin
- Aromatic Electrophilic Substitution: The Preparation of 4-Bromobenzophenone by the Friedel-Crafts Reaction
- The Synthesis of Stilbene: The Wittig Reaction
- Cycloaddition Reactions and their Mechanisms: $4\pi + 2\pi$ and $2\pi + 2\pi$ Reactions Compared
- Sequential Aldol and Michael Addition Reactions followed by Pyridine Synthesis
- Heterocyclic Chemistry: The Guareschi-Thorpe and the Vilsmeier Reactions
- The Preparation and Use of Wilkinson's Catalyst: The Selective Reduction of Carvone
- Chromatography in Action: Comparison of Separation Capabilities of Flash Chromatography, TLC, HPLC, GC and GCMS
- Electrochemistry: Cyclic Voltammetry of a Ferrocene Carboxylic Acid
- Molecular Spectroscopy
- Absorption and Fluorescence of Dye Molecules
- The Stability of Colloidal Particles
- Chemical Kinetics: Stop Flow Methods and the Kinetic Salt Effect
- The Properties of Surfactants
- The Solution Properties of Polyelectrolytes
- Infrared Spectroscopy: Sensitive FTIR to look at Stretching in $^{12}CO_2$ and $^{13}CO_2$
- The Nickel Catalysed Isomerisation of But-1-ene
- Mesoporous Silica formed by Supramolecular Templating and Inorganic Polymers: The Preparation and Properties of Bouncing Putty
- The Preparation of Monomeric and Polymeric Transition Metal Oxides
- The Synthesis and Spectroscopic Characterisation of Metal Carbonyl Complexes
- The Application of Liquified Gases as Non-Aqueous Reaction Media
- Tracing Chlorophyll in Sediments using Chromatography and Visible Spectrophotometry
- Ferrocene: Synthesis and Reactivity
- Preparation of a Luminescent Copper(I) Complex

# PROPOSED NEW THIRD YEAR UNDERGRADUATE LABORATORY COURSE

It is the intention that in the third year, laboratory work will stretch the students much more than in the second year and will require them to manage their own time and give them an element of choice in the experiments they perform. Project-based work and teamwork will also feature and students will be required to make a poster presentation of one of their experiments and to give an oral presentation of another. It is also the intention that students will be challenged with a target molecule and asked to develop their own synthesis using resources available to them. The laboratory will be open for 14 hours a week for 21 weeks.



## List of Experiments

- Transition Metal-Carbon Bonds in Chemistry and Biology
- The Preparation of a Transition Metal Alkylidene Complex
- The Spectroscopic Identification of Unknown Organoruthenium Cluster Compounds
- Homogeneous Hydrogenation Catalysis with Phosphine Complexes of Rhodium
- The Preparation and Structure of the Acetyl Chloride – Antimony Pentachloride Complex
- The Preparation of Carbon-Free Chelate Rings: An Asymmetrical Mixed Donor PNP Ligand and its Palladium Complex
- The Preparation of Carbon-Free Chelate Rings: A Dithioimidophosphinato Ligand and Some of its Metal Complexes
- The Preparation of (2,4,6-Tri-*tert*-butylphenyl)Phosphonous Dichloride
- The Use of the Cambridge Crystallographic Database
- Nickel Complexes of some Schiff Base Ligands
- The Synthesis and Coordination Chemistry of Macrocyclic Complexes
- 2,2':6',2"-Terpyridine Complexes and Metal Directed Reactivity
- Electronic Characterisation of a Transition Metal Complex Using Electrochemical UV/Vis and EPR techniques.
- Synthesis and Characterisation of Colloidal Titania
- Materials Experiment: Using a Surfactant for the Preparation of a Mesoporous Inorganic Material
- Synthesis: A Target Material is Presented to Students who must Devise a Synthesis
- The Determination of the Structures of Palladium(II) Complexes by Infrared and Raman Spectroscopy
- Colloid Science Mini Project. Students Develop a Programme of Experiments with a Variety of Instruments to Test Hypotheses
- Carbon Cycle Mini Project, including, Supercritical $CO_2$, Uptake of $CO_2$ in Droplets and Rotational IR Spectroscopy
- Sharpless Asymmetric Epoxidation
- Alkene Metathesis
- Enolate Formation and Trapping
- The Measurement of the $pK_a$ of Substituted Phenols
- The Synthesis of 18-Crown-6
- Solid Phase Peptide Synthesis
- Photochemical Experiment
- Multistep Reaction Sequence with Palladium-Chemistry as a Key Step
- Carbohydrate Experiment
- Rearrangements in Synthesis
- Stereocontrol in Synthesis
- Aldol Chemistry
- Measurement and Analysis of Respiration

***CONTACT***

Dr PJ Wyatt , Bristol ChemLabS Director and School of Chemistry Director of Undergraduate Studies
tel: +44 (0)117 928 9183, Paul.Wyatt@bristol.ac.uk

Claire-Lise Braun, Secretary to Bristol ChemLabS, tel: +44 (0)117 331 7680, chem-cetl@bristol.ac.uk

# Practical Skill Sets for Undergraduate Chemists

The starting point for designing the new laboratory experiments was to identify the skills we wanted students to have at the end of each year. Many skills are built upon year by year. The skills are outlined below.

## First Year

### Practical
- Identifying & assembling glassware
- What equipment is eg what a Hirsh Funnel looks like
- What kind of containers to use
- Location of laboratory apparatus
- All basic laboratory skills eg recrystallisation
- Making and measuring solutions accurately
- Instrumentation
- Using Vernier scales
- Data-handling and taking observations in a coherent fashion
- Cleanliness of glassware
- Basic synthetic skills
- IR
- TLC

### Safety
- Following instructions
- Risk awareness and working in a safe manner
- Minimising danger to themselves and those around them

### Measurements
- Following detailed instructions - acceptable experimental data
- Recording accurate, reproducible data
- Understanding the importance of testing/calibrating apparatus
- Keeping accurate, detailed laboratory notebooks
- Using software to record data

### Data
- Following instructions to analyse data
- Introduction to using spreadsheets to analyse data
- Demonstrate whether a set of data agrees with a given theory

### Writing
- Presenting data in tabulated and graphical format
- Writing figure legends
- Report writing
- Presenting detailed reports

## Second Year

### Practical
- Distillation
- Dry preparations, reagents-handling
- Handling BuLi
- Small scale synthesis/precious metal handling
- Solid state synthesis
- Gas handling
- Vacuum/inert atmosphere techniques
- Choosing an appropriate technique to address a problem
- Anticipating results - performing / designing experiments accordingly
- Prediction of outcomes as part of experiment planning
- Developing robust experimental protocols
- Understanding the importance of control experiments
- Standard manipulations under inert atmosphere including filtration
- Use of IR to follow a reaction
- Chromatography and HPLC

### Safety
- Basic knowledge of risk assessment - how to minimise risk
- Being aware of COSHH

### Measurements
- Recording accurate, reproducible data
- Understanding the importance of testing and calibrating apparatus
- Understanding how Signal to Noise can be improved
- Keeping accurate, detailed lab-books
- Using software to record data

### Data
- Confidence in using spreadsheets to analyse data
- Demonstrate whether a set of data agrees with a given theory
- Drawing inferences from data sets
- Identifying sources of error in a procedure
- Propagating errors

### Writing
- Use of Crossfire/Web of Science
- Use of Chemical Abstracts
- Use of Aldrich Catalogue
- Writing an abstract
- Giving complete and appropriate references
- Presenting detailed reports for short experiments/ extended projects

## Third Year

### Practical
- Use of low temperatures
- Demanding small scale synthesis (10-20mg)
- Solid state synthesis
- Gas handling
- Standard manipulations under inert atmosphere including filtration
- Advanced vacuum/inert atmosphere techniques
- Choosing an appropriate technique to address a problem in a complex situation
- Anticipating results and performing and/or designing experiments accordingly
- Designing a series of experiments to test a hypothesis
- Use of IR to follow a reaction

### Safety
- Filling in own COSHH form

### Measurements / Data
- Diagnosing problems with malfunctioning apparatus
- Appreciating the strengths and weaknesses associated with “black box” instruments
- Understanding how Signal to Noise can be improved

### Writing
- Advanced use of Crossfire/Web of Science
- Chemical Abstracts Keeping accurate, detailed lab-books

### Transferable Skills
- Presenting experimental results orally
- Preparing a research poster
- Participating in group exercises
- Teamwork